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## In-situ Neutron Diffraction Studies of Various Metals on Engin-X at ISIS

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### 1 Introduction

Pulsed neutron beams available at the ISIS spallation source offer diverse possibilities for materials characterization. ENGIN-X [1] is the dedicated materials engineering neutron beamline at ISIS. The Engin-X diffractometer operates in time-of-flight (TOF) diffraction mode, using neutron pulses with a range of energies which travel a distance of 50 meters towards the sample before being elastically scattered, so that the TOF of a given neutron is proportional to its wavelength. The primary function of the beamline is the determination of residual stresses within the interior of bulk engineering components and test samples, in particular for the development of modern engineering processes (e.g. welding, peening) and variety of structural integrity investigations.

A second important function of the beamline is for studies of fundamental material behaviour, such as composite and rock mechanics, the basic deformation mechanisms of metals, and phase transformations in shape memory alloys and ferroelectrics. In-situ straining neutron diffraction experiments can provide a way to verify assumptions regarding the relative activity of the different deformation systems which can provide valuable information for various polycrystalline deformation models. These models require inputs of the relative activity of the different damage mechanisms operating during deformation. To address this need, a range of sample environment equipment such as hydraulic dynamic mechanical testing rigs, cryogenic [2] and radiant furnaces [3] are available for these investigations on Engin-X as it shown in Table 1.

**Table 1. Range of Engin-X experimental environments setups**

Engin- X experimental setups	Environment	Temp range	Stress rig suitability	Type of tests
Room temperature	Air	20 - 22 °C	50 kN and 100 kN	Compression, tension, fatigue
Cryogenic temperature	Vacuum	-250 - 20 °C		Compression, tension, fatigue
High temperature furnace	Air	20 - 800 °C		Compression, tension, creep, creep-fatigue
High temperature inert gas furnace	Air / Gas ( e.g. Ar)	20 - 1100 °C		

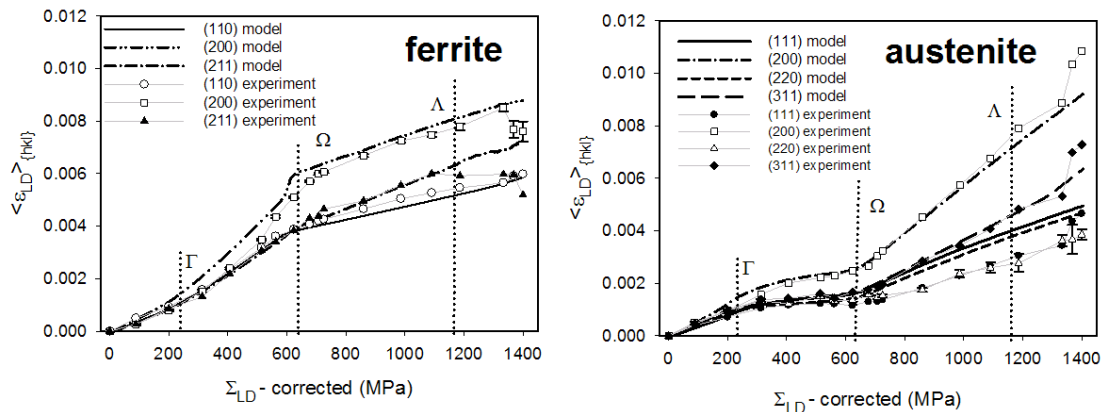
In this paper we present current Engin-X advances in supportive environment and provide examples of applications for in-situ characterization of various industrially relevant metals during mechanical and thermo-mechanical loading.

### 2 Experimental procedure and results

#### 2.1 Room temperature

The instrument is equipped with a 50 kN and 100 kN testing machine mounted on the diffractometer, with its loading axis oriented horizontally at 45° to the incident beam. The two detector banks allow simultaneous collecting of the time-resolved diffraction patterns at fixed horizontal scattering angles of  $\pm 90^\circ$ . The main advantage of the diffraction method is the possibility of studying mechanical properties of polycrystalline materials separately in each phase and in groups of grains with a specific orientation. Due to its selectivity, diffraction is a powerful tool used to analyze the mechanical behavior of polycrystalline materials at the mesoscale (phase and/or grain scale).

In the first example, in-situ of the time-of-flight neutron diffraction during the tensile test and elastoplastic self-consistent modeling were used to study aged stainless duplex steels (50% of ferrite and 50 % of austenite) [4]. Prediction of self-consistent model [5,6] were adjusted to experimental lattice strains  $\langle \varepsilon_{LD} \rangle_{\{hkl\}}$  measured by diffraction for many  $hkl$  reflections vs. stress  $\Sigma_{LD}$  applied to the sample (Fig. 1). The deformation history of samples was divided into parts limited by the characteristic thresholds  $\Gamma$ ,  $\Omega$  ( $\Sigma_{LD} \cong 640\text{MPa}$ ) and  $\Lambda$  ( $\Sigma_{LD} \cong 1150\text{MPa}$ ), determining different ranges of deformation. The positions  $\Gamma$  and  $\Omega$  can be identified as the yield points for the austenitic and ferritic phases, respectively.

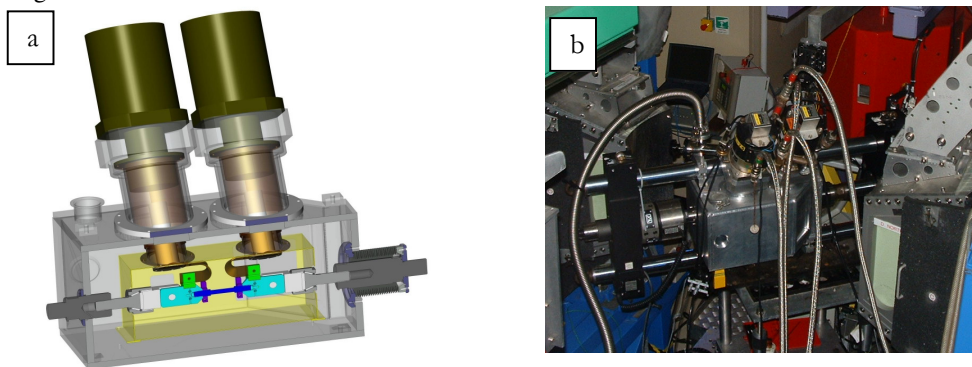


**Figure 1. Elastic lattice strains parallel ( $\langle \epsilon_{LD} \rangle_{\{hkl\}}$ ) to the load direction (LD) versus applied stress  $\Sigma_{LD}$  for aged duplex steel. The results of in situ tensile test (points) are compared with the model predictions (lines).**

The excellent agreement between model calculations and the experimental results up to the threshold  $\Lambda$  confirms that the distribution of stresses between two studied phases is correctly predicted for the elastic and elastoplastic deformation. Model parameters characterizing elastoplastic behavior of both phases (critical resolved stresses and hardening parameters for slip systems) can be determined adjusting theoretical lattice strains  $\langle \epsilon_{LD} \rangle_{\{hkl\}}$  to the experimental ones. Finally, it can be noticed that the differences between experimental results and diffraction data, above the  $\Lambda$  limit, indicate damage phenomena occurring in the studied material (damage is not considered in the present version of self-consistent model).

## 2.2 Cryogenic temperatures

There has been a growing demand for investigations of material mechanical behaviour at cryogenic temperatures due to the recent explosive progress in cryogenic technologies. Applications include cryogenic texture processing of zirconium nuclear alloys, strain sensitivity of superconducting magnet wires, cryogenic structural steels and low temperature shape memory alloys for space applications. In addition to these specific applications, the ability to go down in temperature is generally useful for temperature-dependence studies of deformation modes in metals because of the general increase in yield stress with falling temperature. Thus at lower temperatures data quality is generally improved because of greater elastic strain partitioning, whereas the rapid fall in yield stress of many materials above room temperature tends to result in lower quality, poorly resolved data. The cryogenic stress rig is schematically depicted in Figure 2a.



**Figure 2 Engin-X cryogenic rig a) schematic diagram b) overview of the experimental set up during the experiment.**

Recently the rig was used into investigation of magnesium alloys [7]. Magnesium alloys always exhibit a high directional anisotropy and are hard to deform at room temperature, owing to the hexagonal crystal structure, which limits the modes available for plastic deformation and give rise to significant intergranular stresses. In particular, wrought magnesium alloys usually display a strong in-plane tension-compression asymmetry. This phenomenon has been attributed to the mechanical twinning during compression along the prior working direction. The twins formed by plastic straining can be destroyed again when the sense of loading is reversed. The cyclic twinning-

-detwinning behaviour of magnesium alloys is far from fully understood and attracts lots of attention of experimental researchers and modelers. In this study neutron diffraction measurements were used to quantify the volume fraction of twinning during cyclic loading. Fatigue test sample made from extruded bar of Mg AZ31 were machined with their axes aligned parallel to the extrusion axis. Fig. 2 shows evolution of 00.2 intensity through the cyclic loading at  $-100^{\circ}\text{C}$ . The 00.2 peak emerges at  $-66\text{MPa}$  and keeps increasing with increasing load. Upon unloading the 00.2 peak intensity decreases and then changes the rate of decreasing speed around  $50\text{MPa}$  and finally disappear at  $150\text{MPa}$ , where the untwinning is exhausted and leaves slip as the only available deformation mechanism. This explains the existence of an inflection in the hysteresis stress-strain loop.

### 2.3 High temperatures

The growing interest in properties of materials at high temperatures may be attributed to the dynamic development in technologies where materials are exposed to high temperature environment for example in aero-space industry or fission and fusion nuclear reactors. The Engin-X furnaces (Fig. 3) for neutron scattering measurements of internal stress in engineering materials under load was designed to permit a range of gases to provide non-oxidizing atmosphere for hot samples. The furnace has four focusing infrared elements which are well suited to fit around the sample on the stress rigs (Fig. 4a).

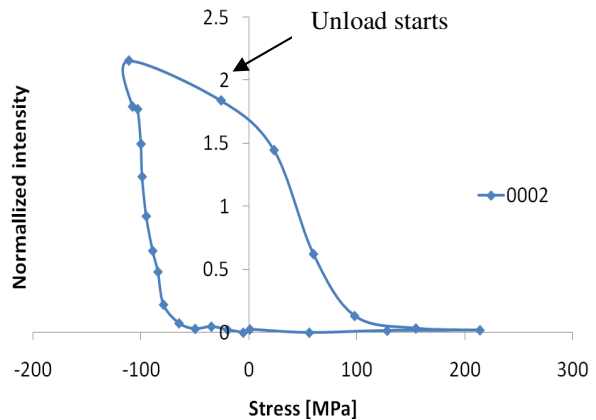
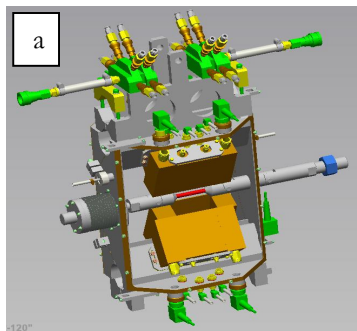


Figure 3. Evolution of 00.2 intensity during cyclic loading

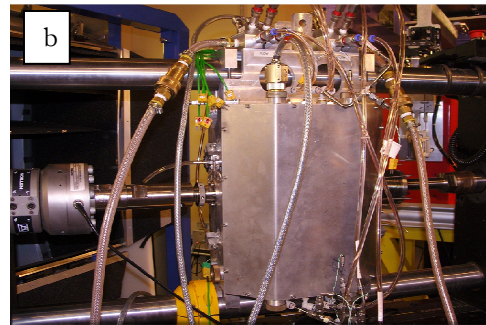


Figure 4. Engin-X inert gas furnace a) Schematic diagram b) overview of the experimental set up during experiment.

Recently, the furnace was used to assess the impact of creep deformation on internal strains in stainless steel samples [8]. Currently, life extensions of a number of power plants are being carried out. Components in these power plants would have undergone a number of stress/temperature cycles which results in complex interactions between deformation modes such as creep and fatigue. The contribution of each of these deformation modes towards internal stresses has not been fully understood. This is vital for safety of critical aging as well as new systems.

In this case it was necessary to correct the data for the pseudo-strains. Correcting the data for d-spacing change occurring due to carbon concentration required looking at one plane which was least influenced by the intergranular strain and then

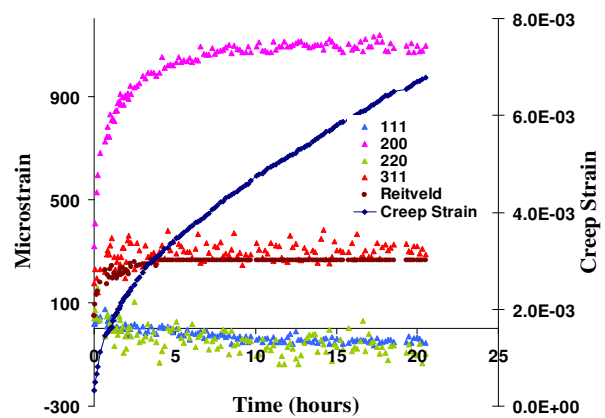


Figure 5. Internal strain data after correcting for elastic lattice strains and changes in carbon concentration

assuming that all the changes seen in that plane is due to changes in carbon concentration. If the strain of one plane is made constant throughout the experiment, then relative differences with the other planes can be used to extract the overall behaviour. The data from the Reitveld refinement or the  $\{311\}$  grain family can be used for this purpose [9]. Fig. 5 shows the data after correction. Fig. 5 shows that during the primary creep phase, there is significant amount of internal strain generation. The  $\{200\}$  and the  $\{311\}$  grain families strain in a tensile manner where as the  $\{220\}$  and the  $\{111\}$  planes strain compressively. The anisotropic behaviour of the grain families thus results in generation internal strains.

### 3 Conclusions

Engin-X beamline is well equipped to study variety of materials in a wide range of temperatures and environments. The ISIS team is constantly working on further improvements of our materials characterization capabilities. For example currently our team is working on implementing the in-situ Engin-X setup into two other beamlines available at ISIS for low angle scattering: LOQ on Target Station One (TS1) and Sans2D Target Station Two (TS2). This new approach will strongly contribute to improving the knowledge and understanding of modern engineering materials.

Additionally, in order to enhance the neutron imaging capabilities at ISIS and to complement the existing materials analysis facilities, the first neutron tomography instrument at a pulsed neutron source is being designed for the ISIS TS2. The new instrument for materials science & engineering imaging, IMAT will be a state-of-the art combined instrument for cold neutron radiography and diffraction analysis for materials science, materials processing, and engineering studies. The instrument will provide the largest possible neutron flux available for imaging at ISIS and will allow medium-resolution neutron “colour” imaging and diffraction (strain scanning and texture during one experiment). The ability to perform imaging and diffraction studies on the same beamline with a single sample set-up will offer unprecedented opportunities for a new generation of neutron studies [10].

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